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Experimental Procedure and Results for Contact Thermal Conductance Measurements performed during FY 2021

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Introduction

Weapons Test Engineering groups at LANL are responsible for testing and simulating weapon assemblies and subassemblies in support of qualification and certification of nuclear weapon systems. The complex nature of the assemblies themselves coupled with the loading environments, gives rise to low confidence and high uncertainties in the interpretation of both experimental and simulated results. In an effort to reduce these uncertainties, a project was started at LANL to perform focused and systematic experiments combined with simulations to increase confidence in both arenas. A key goal of the work performed under the Delivery Environments (DE) Testbeds to Reduce Uncertainties in Simulations and Tests TRUST program, focuses on identifying and minimizing sources of uncertainty associated with experimental and computational techniques. Specifically, with the above as motivation, a measurement procedure was developed by MST-8 to perform thermal conductivity measurements on cylinders of both similar and dissimilar metallic systems as a function of loading conditions and varying material interface morphologies between these components. The title of the work package for this work is "Contact Thermal Conductance (CTC)". The main goal of the measurements performed under the 2021 CTC work package was to apply the procedures developed under the 2020 work package and perform measurements to provide a dataset that can be used to test W-13 models currently being developed. Specifically, the CTC measurements were performed while material were under constant loads of 15, 500 and 5000N, in triplicate, for 3 different material combinations: 304L Stainless Steel-304L Stainless Steel, Aluminum-Aluminum and 304L Stainless Steel-Aluminum. The interfaces of the materials tested consisted of 2 different nominal targeted surface finishes of 0.8 and 1.6 microns, the measured finishes will be listed in a later section. A single targeted temperature gradient (approximately 20°C) across the experimental assembly was initially adjusted in the 15 N loading condition and the cold side of the experiment fixed at -75°C based on the cryogenic controller for all measurements. Tuning of the targeted temperature gradient across material interface was done via controlling the heated

side of the test assembly thereby keeping the variables at a minimum. These parameters were fixed and used in the subsequent measurements using forces of 500 and 5000N, respectively. The results obtained from these measurements will be used to validate predictive models and validation simulation efforts with a focus on uncertainty. In this report henceforth, the experimental methodology developed in FY 2020 will be applied, although the measurements have been relocated to a new dedicated test system to measure thermal conductivity across material interfaces.

Specimen preparation for measurements

1. Experimental specimen details and machining

The specimens for conductivity measurements were obtained from rods of certified Valbruna 304L stainless steel (SS) and Hydro 6061 Aluminum (Al) base materials. Certification material test reports (CMRT) for each material are attached in the supplementary section of this report. Specimens for CTC measurements were machined as right cylinders with nominal dimensions of 100 mm in length and 25.4 mm in diameter. Holes were drilled to specific depths of 12.7 mm for the thermocouples along the length of the rods at the specified locations as shown in Fig. 1. The thermocouple (Tc) hole spacing is indicated by hole center marks "+" and the hole depths are represented by dotted lines in Fig. 1. Specific surface finishes of 1.6 µm and 0.8 µm were machined on the sample interface ends. The nominal dimensional specifications requested for machining are shown in Fig. 1.

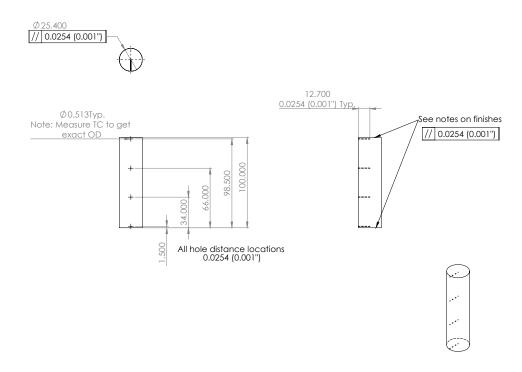


Figure 1. Machine drawing showing the nominal geometric specifications for test specimens, dimensions are in mm. For clarity, critical tolerances are called out in both mm and in.

After machining, the CTC specimen's Tc spacing were carefully measured using a tool maker's microscope and the final attained surface finish was measured using a Mahr Mobile Surface Roughness Tester, model MarSurf PS10 to provide an accurate representation of the samples to decrease uncertainty in the predictive modeling efforts. Fig. 2. shows a diagram of the three CTC specimen combinations Tc spacing along with Table 1 which lists the Tc spacing and the Tc distance across the samples with respect to the cold and hot interface.

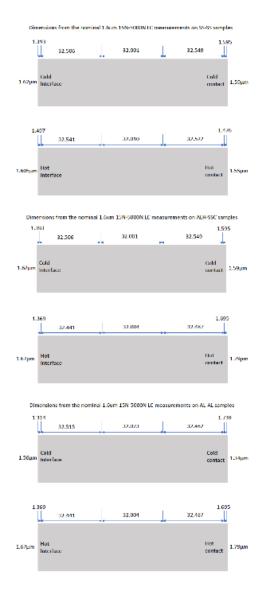


Figure 2. Measured Tc spacing for the nominal 1.6μm surface finish SS-SS, Al-Al and Al-SS test specimens used for CTC measurements.

Table 1: Measured Tc spacing's and Tc distances from the cold and hot interface for the three nominal 1.6µm interface surface finish test specimen series

	304L Stainl	ess Steel Cold	(mm)	
cold interface to TC6	TC6 to TC5	TC5 to TC4	TC4 to TC3	TC3 to cold platen
1.39	32.51	32.00	32.55	1.60
cold interface (CI) to TC6	CI to TC5	CI t to TC4	CI t to TC3	CI to cold platen
1.39	33.90	65.90	98.45	100.04
	304L Stain	less Steel Hot	(mm)	
hot interface to TC7	TC7 to TC8	TC8 to TC9	TC9 to TC10	TC10 to hot platen
1.50	32.54	32.01	32.52	1.48
hot interface (HI) to TC7	HI to TC8	HI to TC9	HI to TC10	HI to hot platen
1.50	34.04	66.05	98.57	100.05
	Aluminur	n 6061 Cold (1	mm)	
cold interface to TC6	TC6 to TC5	TC5 to TC4	TC4 to TC3	TC3 to cold platen
1.31	32.51	32.02	32.46	1.74
cold interface (CI) to TC6	CI t to TC5	CI t to TC4	CI to TC3	CI to cold platen
1.31	33.83	65.85	98.31	100.05
	Aluminu	m 6061 Hot (n	nm)	
hot interface to TC7	TC7 to TC8	TC8 to TC9	TC9 to TC10	TC10 to hot platen
1.37	32.44	32.00	32.49	1.70
hot interface (HI) to TC7	HI to TC8	HI to TC9	HI to TC10	HI to hot platen
1.37	33.81	65.81	98.30	100.00
	304L Stainl	ess Steel Cold	(mm)	
cold interface to TC6	TC6 to TC5	TC5 to TC4	TC4 to TC3	TC3 to cold platen
1.39	32.51	32.00	32.55	1.60
cold interface (CI) to TC6	CI to TC5	CI to TC4	CI t to TC3	CI to cold platen
1.39	33.90	65.90	98.45	100.04
	Aluminu	m 6061 Hot (n	nm)	
hot interface to TC7	TC7 to TC8	TC8 to TC9	TC9 to TC10	TC10 to hot platen
1.37	32.44	32.00	32.49	1.70
hot interface (HI) to TC7	HI to TC8	HI to TC9	HI to TC10	HI to hot platen
1.37	33.81	65.81	98.30	100.00

Premeasurement setup

1. Experimental setup

The procedure for the measurement setup is discussed in the following sections in the order they were performed: mounting of the test specimens in the experimental measurement setup (consisted of a MTS 858 Mini Bionix II® load frame with heated and cooling platens), instrumentation of the experimental test specimens with temperature probes and lastly, application of insulation around the test specimens prior to application of the temperature gradient. Much of the precise details were omitted in this report in the fact that they were discussed in detail in the previous report. Only new details pertaining to the 2021 system and associated measurements will be discussed as needed. A 16-channel Omega thermocouple data logger (model OM-DAQXL-2-EU8) using Omega k-type

thermocouples (model TJC36-CASS-020U-6) was used to log the temperature of the specimens and collect the raw load vs. displacement signals during measurements. After the measurements on the 304L SS specimens were completed, the original Omega thermocouple (Tc) data logger malfunctioned and was replaced with an almost identical data logger (model OM-DAQXL-2-NA). To apply the load along the experimental specimen, a MTS 858 servo-hydraulic test system equipped with a resistance heated upper platen and a liquid nitrogen cooled lower platen was used. In contrast to the previous system, the current mechanical test frame applied the load from the top down in contrast to the first-generation CTC setup. Next, the Tc's were placed into the predrilled locations using silver paste to ensure good contact and increase system responsiveness. The Tc's were marked with fiducials corresponding to the callout hole depths. Special care was taken to make sure the Tc's were inserted down into the entire hole depth to maintain reproducibility for simulations. Finally, insulation was applied around the test specimens. A generic setup photo including instrumented samples in position and under load along with a schematic listing of key components is shown in Fig. 3.

Heated Platen controller Heated platen Samples Water recirculatory Data logger

2021 CTC TRUST setup

2021 CTC TRUST setup and sample schematic

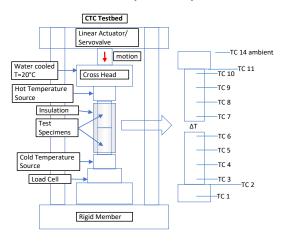


Figure 3. A photo of the experimental setup showing details prior to application of insulation. The experimental testbed ready for measurements is shown schematically on the rightmost portion of the image.

Cryo-controller

Experimental measurement

2. Force and temperature gradient establishment

Following the pre-measurement setup discussed previously, the next step is preparing the specimens for the respective experiments. One thing to note, the lowest force of 0.03 MPa (15N) corresponded to the least applied force that would provide enough thermal contact between the heating/cooling platens and samples while under stable load control from the MTS 858 system. A flowchart showing the experiment measurement work flow is shown in Fig 3.

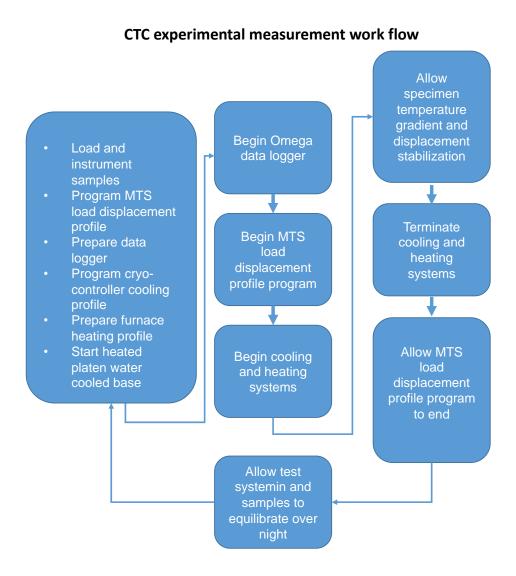


Figure 4. Flowchart showing the typical CTC experimental measurement work flow for performing CTC measurements.

Post-Measurement

Upon completion of the CTC test measurements, data was compiled and prepared for plotting. To archive and have a repository for viewing the tests performed and uploaded to TIMS, temperature gradient along the samples (°C) vs. time (s) and force (N)/displacement (mm) (FD) vs. time (s) plots for SS-SS, Al-Al and Al-SS couples are presented side by side, in groups of three, based on the individual loading conditions of 15,500 and 5000N used. The plots presented are grouped by loading condition for temperature gradient evolution versus time and show a full measurement range with all the sample Tc signals along with a magnified region below. The magnified lower plot shows the region with the two Tc's closest to the cold and hot interface where C6 is the Tc closest to the cold interface and C7 is the Tc closest to the hot interface as indicated and circled in Fig. 5.

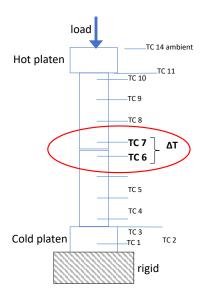


Figure 5. Schematic showing the Tc names and locations used to measure the temperature gradients along the sample lengths. The circled region shows Tc C6 and C7 which are the sensors measuring the ΔT across the hot and cold side interface.

Pertaining to the magnified plots for both sets (Tc's and FD) the magnified regions show a snapshot of the dwell stabilization period about 500 s before the abrupt behavior signaling when the cooling and heating sources were instantaneously turned off. The point in the measurements when the heating and cooling was turned off varied from measurement to

measurement. Similar to the Tc vs time plots, the FD plots are shown as the full data range on top with a magnified view below highlighting the region before the cooling and heating sources were turned off. The Y1 axis is force (N) and the Y2 is displacement (mm), with the force signals circled in the magnified view to easily distinguish it from the displacement signals. In both sets of plots the period in time showing the magnified regions is outline by a box on the full plot versions. The main regions of interest in these measurements for W-13 modeling and simulations efforts was achieved at a point in the measurement when the temperature had equilibrated and the specimens were undergoing a cumulative change in displacement of 2 μ m or less. It is in this final region before turning off the heating and cooling where W-13 calculations will be targeted as highlighted in the following magnified plots. In the follow pages, Figs. 6,7,8 (a-c) are plotted as follows:

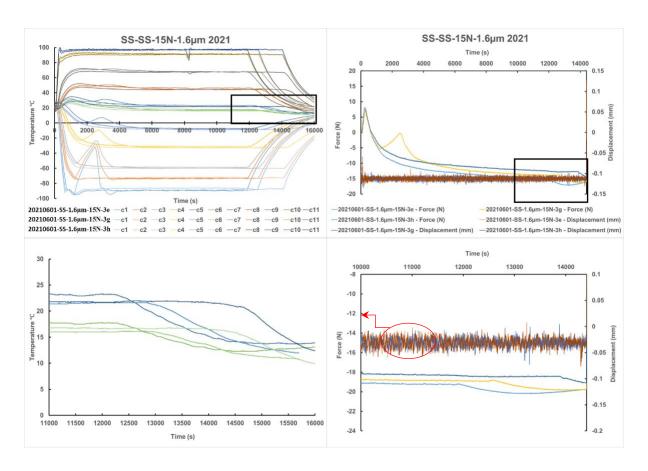


Figure 6a. Temperature as a function of time corresponding to 15N force between SS-SS.

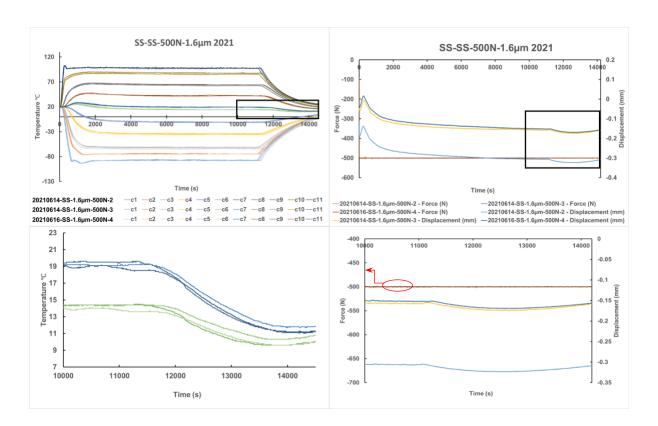


Figure 6b. Temperature as a function of time corresponding to 500N force between SS-SS.

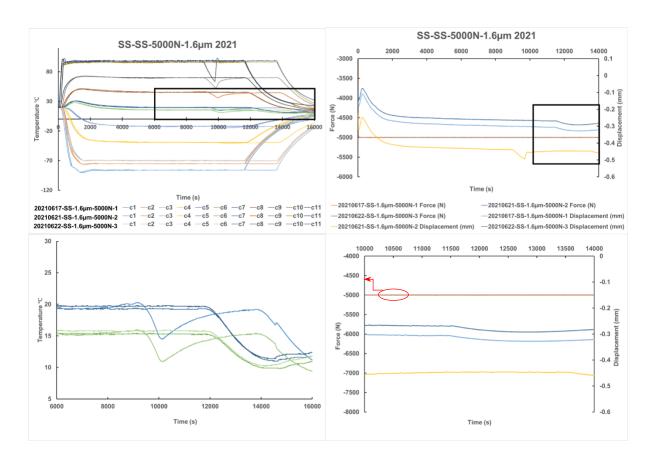


Figure 6c. Temperature as a function of time corresponding to 5000N force between SS-SS.

Figures 6 a-c. Measurements plotted in triplicate for SS-SS specimens with nominal 1.6µm surface finish. The upper left plots are the full range Tc measurements (°C) vs. time with the lower left showing a magnified view before the heating and cooling sources and terminated. The upper right most plots show load (N) and displacement (mm) vs. time(s). Figures 6 a-c correspond to 15, 500 and 5000 N loading conditions. A magnified view for each of the upper plots are shown below them.

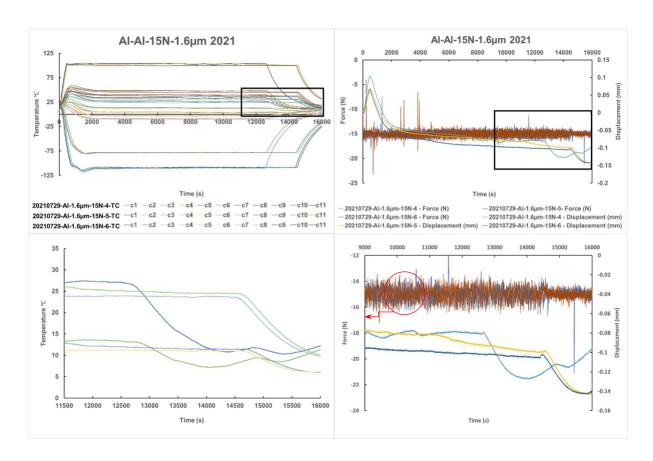


Figure 7a. Temperature as a function of time corresponding to 15N force between Al-AL.

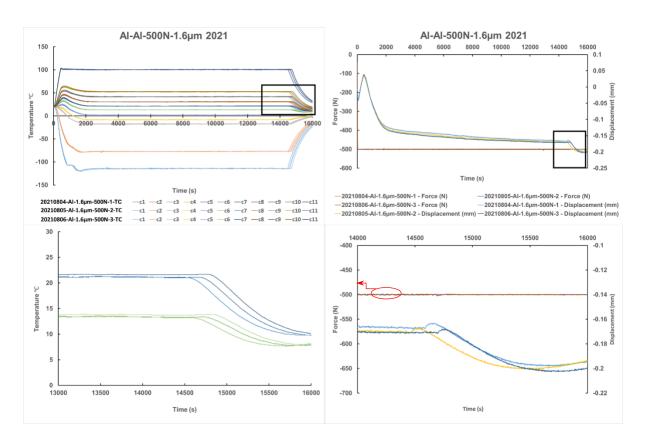


Figure 7b. Temperature as a function of time corresponding to 500N force between Al-AL.

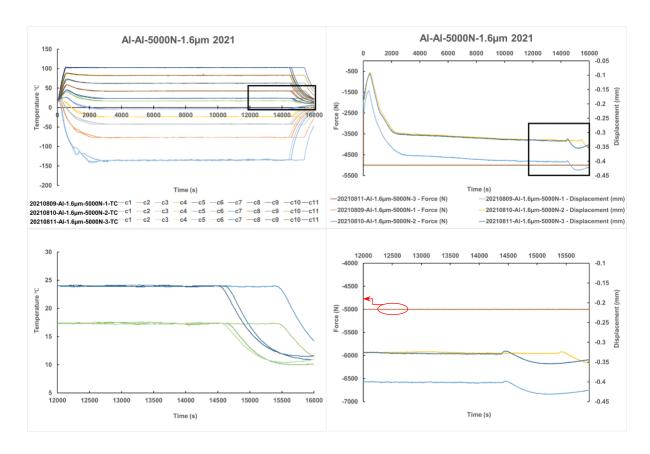


Figure 7c. Temperature as a function of time correspond to 5000 N force between Al-AL.

Figure 7 a-c. Measurements plotted in triplicate for Al-Al specimens with nominal 1.6μm surface finish. The upper left plots are the full range Tc measurements (°C) vs. time with the lower left showing a magnified view before the heating and cooling sources and terminated. The upper right most plots show load (N) and displacement (mm) vs. time(s). Figures 7 a-c correspond to 15, 500 and 5000 N loading conditions. A magnified view for each of the upper plots are shown below them.

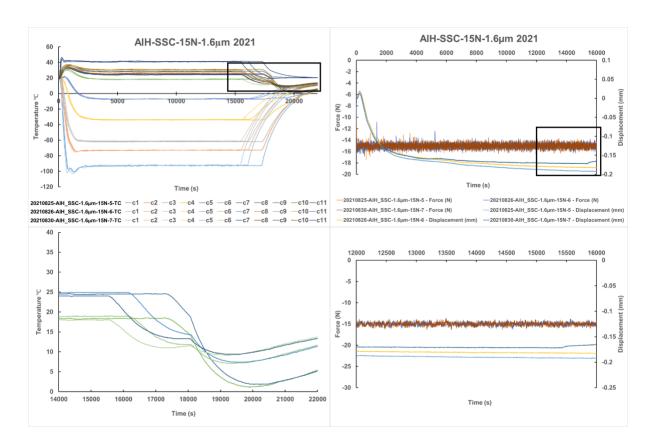


Figure 8a. Temperature as a function of time correspond to 15 N force between AlH-SSC.

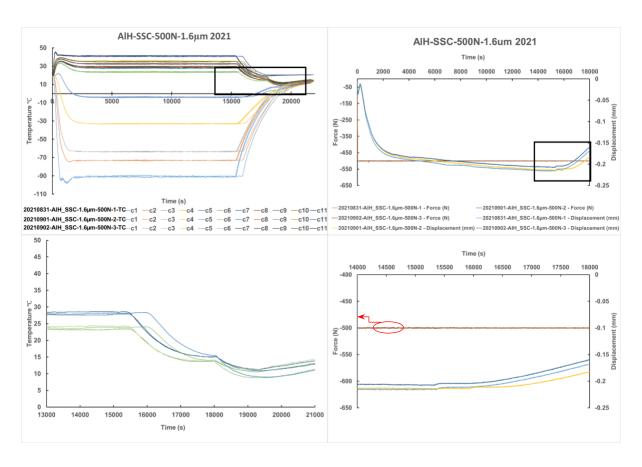


Figure 8b. Temperature as a function of time correspond to 500 N force between AlH-SSC.

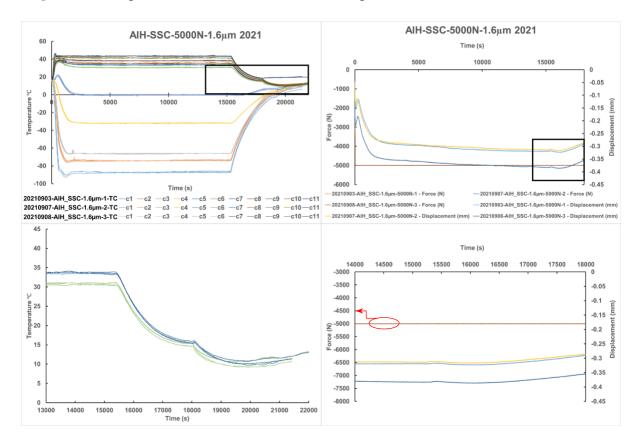


Figure 8c. Temperature as a function of time correspond to 5000 N force between AlH-SSC.

Figure 8 a-c. Measurements plotted in triplicate for AlH-AlC specimens with nominal 1.6μm surface finish. The upper left plots are the full range Tc measurements (°C) vs. time with the lower left showing a magnified view before the heating and cooling sources and terminated. The upper right most plots show load (N) and displacement (mm) vs. time(s). Figures 8 a-c correspond to 15, 500 and 5000 N loading conditions. A magnified view for each of the upper plots are shown below them.

In the early measurements (SS-SS), some aberrations were present in measurement plots in the path to establishing a temperature gradient. The sources of aberrations were due to a faulty temperature controller and problems with facility cooling system. Although these aberrations are not pleasing to data, the period when they occurred are not in the time-frame where data is extracted for analysis. These problems were rectified, as is apparent in the stability of the measurements as they continued. Also, to note in the early tests there was some iterative changes in the initial parameters for the temperatures used to establish the temperature gradients, after which consensus in the setup parameters was finalized and maintained through future measurements. To illustrate the consistency in measurements performed in triplicate, Table 2. shows the Tc temperature signals from C6 and C7 (taken from 500 s before the cooling and heating was turned off) and the average calculated temperature between them and across the interface.

Table 2: To measurement data at steady state showing the average temperature in between the two To's above and below the interface, C6 and C7.

Al-AL	C6 Temperature °C	C7 Temperature °C	average ΔT across interface °C
20210729-Al-1.6um-15N-4-TC	13.6	27.2	20.4
20210802-Al-1.6um-15N-5-TC	11.5	24.5	18
20210602-Al-1.6um-15N-6-TC	11.1	23.7	17.4
20210804-Al-1.6um-500N-1-TC	13.2	21.1	17.15
20210805-Al-1.6um-500N-2-TC	13.3	21.1	17.2
20210806-Al-1.6um-500N-3-TC	13.7	21.6	17.65
20210809-Al-1.6um-5000N-1-TC	17.1	23.8	20.45
20210810-Al-1.6um-5000N-2-TC	17.2	23.9	20.55
20210811-Al-1.6um-5000N-3-TC	17.2	23.9	20.55
SS-SS	C6 Temperature °C	C7 Temperature °C	ΔT across interface °C
20210601-SS-1.6um-15N-3e	17.6	23	20.3
20210601-SS-1.6um-15N-3h	16.7	21.8	19.25
20210601-SS-1.6um-15N-3g	16.1	21.6	18.85

20210614-SS-1.6um-500N-2	14.4	19.5	16.95
20210614-SS-1.6um-500N-3	14.4	19.1	16.75
20210616-SS-1.6um-500N-4	14.3	19.6	16.95
20210417 57 4 5 700004	47.0	10.0	15.05
20210617-SS-1.6um-5000N-1	15.3	19.2	17.25
20210621-SS-1.6um-5000N-2	15.2	19	17.1
20210622-SS-1.6um-5000N-3	15.8	19.6	17.7
	C6 Temperature	C7 Temperature	ΔT across interface
AlH-SSC	°C	°C	°C
20210825-AlH_SSC-1.6um-15N-5-TC	18.4	24.6	21.5
20210826-AlH_SSC-1.6um-15N-6-TC	18.9	24.9	21.9
20210830-AIH_SSC-1.6um-15N-7-TC	18	24	21
20210831-AIH_SSC-1.6um-500N-1-TC	23.3	27.7	25.5
20210901-AIH_SSC-1.6um-500N-2-TC	23.8	28.2	26
20210902-AIH_SSC-1.6um-500N-3-TC	24.3	28.6	26.45
20210903-AIH_SSC-1.6um-5000N-1-TC	30.9	33.8	32.35
20210907-AIH_SSC-1.6um-5000N-2-TC	30.6	33.6	32.1
20210908-AIH_SSC-1.6um-5000N-3-TC	30.5	33.4	31.95

As a deliverable for the CTC project, measurement data was required to be uploaded into the GRANTA/TIMS database for access by project members. Below is a table with the completed measurement dataset uploaded into the database.

Table 3: Summary of measurements performed and files uploaded into GRANTA/TIMS

CTC measurement files						
Stainless Steel	-Stainless Steel	Aluminum	-Aluminum	Aluminum H-S	Stainless Steel C	
load vs displacement	Temperature, load vs displacement (raw)	load vs displacement	Temperature, load vs displacement (raw)	load vs displacement	Temperature, load vs displacement (raw)	
15N	15N	15N	15N	15N	15N	
20210601-SS- 15N-3b	20210601-SS- 15N-3b-tc	20210729-Al- 16um-15N-4	20210729-Al- 16um-15N-4-tc	20210824- AlH_SSC-16um- 15N-4	20210824- AIH_SSC-16um- 15N-4-tc	
20210601-SS- 15N-3c	20210601-SS- 15N-3c-tc	20210729-Al- 16um-15N-5	20210729-Al- 16um-15N-5-tc	20210824- AIH_SSC-16um- 15N-5	20210824- AIH_SSC-16um- 15N-5-tc	
20210601-SS- 15N-3d	20210601-SS- 15N-3d-tc	20210729-Al- 16um-15N-6	20210729-Al- 16um-15N-6-tc	20210824- AIH_SSC-16um- 15N-6	20210824- AIH_SSC-16um- 15N-6-tc	
20210601-SS- 15N-3e	20210601-SS- 15N-3e-tc			20210824- AIH_SSC-16um- 15N-7	20210824- AIH_SSC-16um- 15N-7-tc	
20210601-SS- 15N-3g	20210601-SS- 15N-3g-tc					
500N	500N	500N	500N	500N	500N	
20210611-SS- 16um-500N-1 20210611-SS-	20210611-SS- 16um-500N-1-tc 20210611-SS-	20210804-Al- 16um-500N-1 20210804-Al-	20210804-Al- 16um-500N-1-tc 20210804-Al-	TRST ALH-SSC 500N 1.6um-1 TRST ALH-SSC	TRST ALH-SSC 500N 1.6um-1-tc TRST ALH-SSC	
16um-500N-2	16um-500N-2-tc	16um-500N-2	16um-500N-2-tc	500N 1.6um-2	500N 1.6um-2-tc	
20210611-SS- 16um-500N-3	20210611-SS- 16um-500N-3-tc	20210804-Al- 16um-500N-3	20210804-Al- 16um-500N-3-tc	TRST ALH-SSC 500N 1.6um-3	TRST ALH-SSC 500N 1.6um-3-tc	
20210611-SS- 16um-500N-4	20210611-SS- 16um-500N-4-tc					
5000N	5000N	5000N	5000N	5000N	5000N	
20210617-SS- 16um-5000N-1 20210617-SS- 16um-5000N-2	20210617-SS- 16um-5000N-1-tc 20210617-SS- 16um-5000N-2-tc	20210809-Al- 16um-5000N-1 20210809-Al- 16um-5000N-2	20210809-Al- 16um-5000N-1-tc 20210809-Al- 16um-5000N-2-tc	TRST ALH-SSC 5000N 1.6um-1 TRST ALH-SSC 5000N 1.6um-2	TRST ALH-SSC 5000N 1.6um-1-tc TRST ALH-SSC 5000N 1.6um-2-tc	
20210617-SS- 16um-5000N-3	20210617-SS- 16um-5000N-3-tc	20210809-Al- 16um-5000N-3	20210809-Al- 16um-5000N-3-tc	TRST ALH-SSC 5000N 1.6um-3	TRST ALH-SSC 5000N 1.6um-3-tc	

Summary

For the FY 2021 CTC project MST-8 was able to further expand and improve the capability to apply and measure a set temperature gradient along the length and across the interface of a test specimen couple while applying a controlled force. During the current project year all 4 quarter deliverables were met and are shown below:

Table 3: CTC project quarterly deliverables and status

Thermal Contact Conductance (CTC) 2021 quarterly de	liverables
Q1. Experiments of expanded material and boundary condition test matrix with results uploaded to TIMS (FY21 Q1) MST-8	Completed and reported

Q2. Validation simulations including UQ propagation from experimental measurements (FY21 Q2) W-13	Completed and reported
Q3. Validation experiments with updated boundary conditions with results uploaded to TIMS (FY21 Q3) MST-8	Completed and reported
Q4. Simulation and experimental results comparison and reports (FY21 Q4) W-13/MST-8	Completed upon transmission of this report

The highlight accomplishments we would like to call attention to are: A predicted displacement control profile obtained from W-13 simulation results on 304L SS was run by MST-8 in the measurement platform, this process revealed more work was required to accurately transition from a simulation predicted profile to measured profiles (discussed in detailed by W-13 in their FY 2021 report). This example shows the strong collaborative and iterative work between W-13 and MST-8. In FY 2021, a complete dataset of 27 measurements were performed and uploaded to the TIMS database using fixed temperature gradient profiles, a manuscript by Ben-Naim T. et al. is in preparation showing mechanical property test data from this project. Lastly, there was strong utilization of GRANTA/TIMS between the MST-8 and W-13 CTC team.

Supplementary information

1. Certification sheets for materials used in mechanical properties (including OFHC) and CTC measurements

6061 Aluminum

Hydro

Invoice To Customer

BRALCO METALS 6718 JEFFERSON NE

6718 JEFFERSON N.E.

BRALCO METALS

ALBUQUERQUE, NM - 87109

ALBUQUERQUE, NM - 87109

Hydro Extrasion USA, LLC 1550 N. Hydro Way

SPANISH FORK, UT

1101584023 Sales Order Number

Certified Test Report

Line No. 11-53991-1 Customer P/O 26-OCT-19

HYDRO3150133 Cert Number Cert Creation Date Page Cert Print Date Page 1 of 3

26-OCT-19

Delivery Id BAL CustomerPart No. applicable Specifications, Revisions and Exceptions 5147534 964 Quantity Shipped IAR 1056328 LB Item No. Date Shipped Item No. Rev 26-OCT-19 G03967449 Item Description ACC-U-LINE Extruded ACC-U-ROD 1,000 DIA 4+,003 SECT 555336 144,000 IN LN FIN M-MILL W/F 924 F3 CS I 606/J76511H Marking CONTINUOUS; Specification
ASTM B221 REV 14
Table 1 Chemical Composition Limits
Table 2 Mechanical Property Limits
ASME SB 221 REV 17
Table 1 Chemical Composition Limits
Table 1 Chemical Composition Limits
Table 2 Mechanical Properties Limits
AMS QQ-A-2008 REV A

pecifications referenced herein for the extrusions subject to this certified test report, although Hydro Extrusion USA, LLC (Hydro) complies with the material requirements of ASTM product

ASTM B241/B241M and ASME SB 241/241M. ASMESB 221 and AMS QQ-A-200/8A, Tables I through 4 for ASTM B317/B317M and Tables 1 and 3 for aluminum extrusions, Hydro follows its own standard processes to achieve applicable customer-specific and ASTM product requirements. Only those specifications (identified above) are and methods. While Hydro generally follows the ASTM-accepted process routing for manufacturing certified to the following tables (excluding footnotes) Tables 1 and 2 for ASTM B221, B429/B429M, luding processes related to temperature, time, frequency, speed, and other process parameters Hydros practices may not align with certain processes referenced in those specifications,

COMPOSITION NOTE: The values for 'Others Each' and 'Others Total' have met the limits as shown on American Act (DFARS 252.225-7001) American Act DFARS 252.225-7001 Melted in the USA or a qualifying country, as identified in the Buy or U.S. Government Sales (when applicable to extrusions): Items supplied are compliant with the Buy

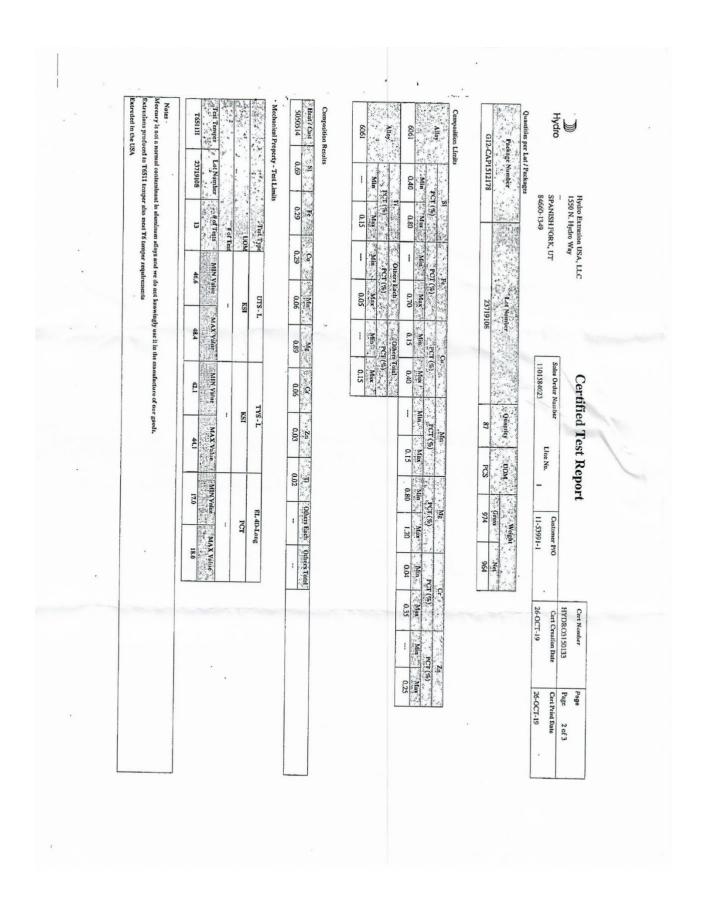
his certified inspection report. Remainder is Aluminum.

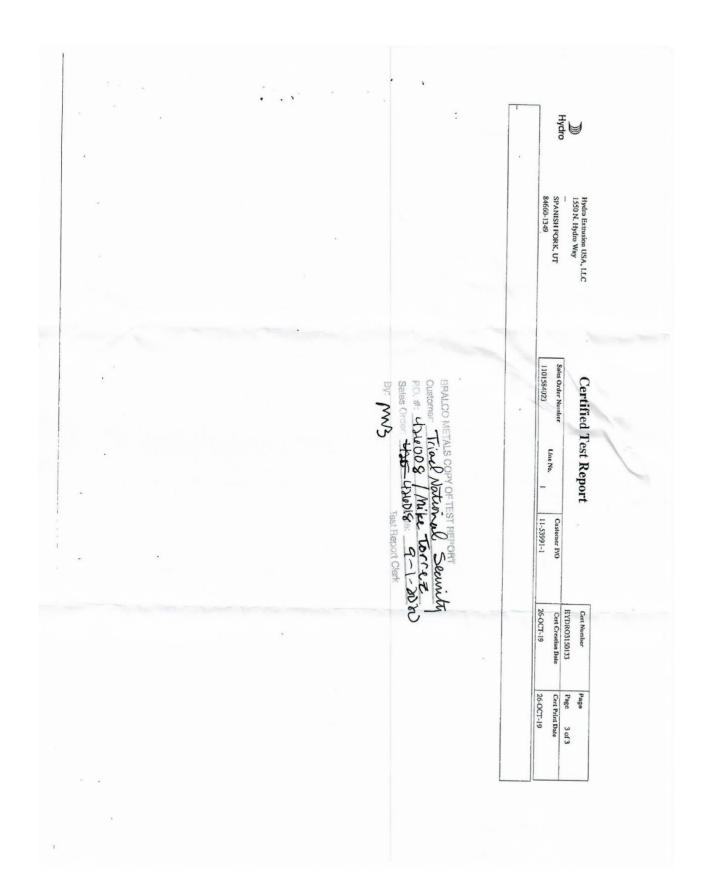
Signature And Title

Hydro Extrusion USA, LLC hereby certifies that the extrusions covered in this report are within the acceptable ranges of the sections or the specification tables, excluding foundes, identified herein, Chemical composition may be based on results provided by external billet, suppliers. Further information on processing, testing including ASTM B557, and inspection is available at www.hydro.com/technicalspecificationsENA. Sales are governed by the Extrasion North America Standard Terms and Conditions of Sale available at www.hydro.com/termsconditionsENA, unless otherwise mutually agreed in writing.

Steven Tanner Quality Manager

26-OCT-19





304L Stainless Steel



SLATER STAINLESS, INC. 200 TAYLOG STATES WEST PORT WATER STAINLESS, INC. Report Number: 5633502
Order LD Order Date Commodity Code
OA19000711 0040 4/30/19 304D1.0
1.0000 275445 0008000061 63166
Round Cold Drawn Finish 300/2007
144.000 Min. 156.000 Max. PL19001445/1 005621 LBS
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Ship 8105 BREEN ROAD 2400 TAYLOR STREET WEST
To HOUSTON, TX 77064 To FORT WAYNE, EN 46802
Lifts: 0004 0005 Condition A
*ASME SA182 13 Chem Only ASME SA479-13
AMS 5647K SAE-AMS QQ-S-763D
CHEMICAL ANALYSIS (Weight %) C Mn P S Si CF N4 W
O12 1.86 030 024 58 18.15 8.09 45 51 09 BRINELL Type A device E10-18
HBW 239
ROOM TEMP TENSILE ASTM ER-169
TS (PSI) .2%YS (PSI) %EL (4d) %RA 107000 85100 42.7 74.4
MACRO ASTM E340-15/E381-17 MACRO
GRAIN SIZE ASTM E112 13
GRAIN SIZE Etchant Magnification X
ASTM A262-15 PR E(A Utilized)
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Material when shipped is free from contempation by
Electric Furnace melted AOD refined
*Solution annealed at 1940 dec From hour and water
*Cert revised @ VSSI by A.Asiala 10/11/2019 Chemical testing to one or more of the listed ASTM methods:
E1017-10, E4080-14
Location of tensile fracture: inside middle of gage length: %elong after fracture. Chemical Analysis performed at Acciaierie Valbruna; Italy.
Results relate only to the items tested. Certification shall not be reproduced except in full, without written approval of Valbruna Stainless inc. The recording of false, fictitious, or fraudulent statements on this document may be punished as a felony under rederal statutes, including Federal law Title 18, Chapter 47. Consult material safety data sheet (MSDS) for hazard info. hereby certify that the reported figures are correct as contained in the records of the corporation.
Manager Laboratory Services Q Q Q Q
emait: AAşfala@valbruna.us Annmarie C Asiàfa



Product Certification Report

Report Number: 5633502

Order D. Certifie	d on May 03, 2019 Page 2 of 2
	2013 Page 2 Of 2
OA19000711 0040 4/30/19 304D1.0	
DIM 1 DIm 2 Heat I.D. Customer I.D.	Customer Purchase Order
1.0000 275446 0008000061	63166
Product Shape Product Surface	02100
- I demonstrate the second to	Qustomer Grade
Legath (Inches)	304/304E
Bill of Lading #	
144.000 Min. 156.000 Max. PL19001445	/1 005621 LBS
()	T COSOZI LBS

VALBRUNA STAINLESS, INC.
Ship 8105 BREEN ROAD
HOUSTON, TX 77064

VALBRUNA STAINLESS INC. 2400 TAYLOR STREET WEST FORT WAYNE IN 46802

No mercury or low melting alloy contamination, No weld repair.

Material melted in Italy, manufactured in the United States.

Material conforms to listed specifications. DFARS compliant. Our heat treat furnaces use air atmosphere.

Quality system is compliant with ISO 9001:2015. Produced in accordance with EN 10204 3.14

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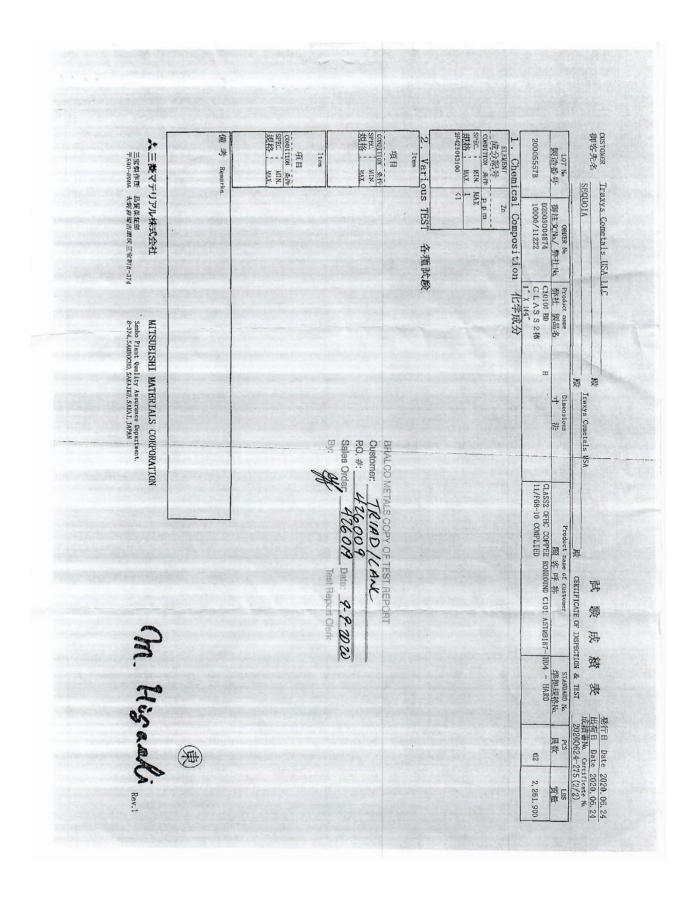
Results relate only to the items tested. Certification shall not be reproduced except in full, without written approval of Valbruna Stainless Inc. The recording of false, fictilitous, or fraudulent statements on this document may be punished as a felony under federal statutes, including Federal law Title 18, Chapter 47. Consult material safety data sheet (MSDS) for hazard info. I hereby certify that the reported figures are correct as contained in the records of the corporation.

Manager Laboratory Services

email: AAsiala@valbruna.us

Annmarie C Asiala

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2. Thermal Conductivity Measurements

A Transient Plane Source method using a Hot Disk 2500 S Thermal Constants Analyzer was used to measure thermal conductivity of 304L stainless steel, Aluminum 6061, and Oxygen Free Copper (OFHC) Cu with certified material test reports (attached at the beginning of this section) using a Kapton Sensor 5465 [1]. Identical tests were performed on none certified materials and can be seen in the FY 2020 version the CTC year-end report. The details of the system used are listed in Table I.

Table I: Hot Disk 2500 Transient Plane Source Specifications

Hot Disc	2500	
1	Thermal Conductivity	0.005 to 1800 W/m/K
2	Thermal Diffusivity	$0.01 \text{ to } 1200 \text{ mm}^2\text{/s}$
3	Measured Time	1 to 2560 seconds
4	Accuracy	Better than 5%
5	Temperature Range	-253°C to 1000°C
6	With Furnace	Up to 750°C [1000°C oxygen free]
7	With Circulator	-35°C to 200°C
8	Power requirement	Adjusted to the line voltage in the country of use
9	Smallest sample dimension	0.5 mm x 2 mm diameter of square for bulk testing
	_	0.042 mm x 8 mm diameter or square for slab testing
		5 mm x 2.5 mm diameter or square for one-dimensional testing
		0.01 mm x 22 mm diameter or square for thin-film testing
10	Sensor Type	All Kapton, All Mica, All Teflon

Tests were conducted by applying power in watts and holding it constant for a few minutes during room temperature tests and up to 15 minutes for 200°C measurements. The 5465 sensor was held between two identical cylinders of the test material with an estimated 0.247 Nm [2] (finger tight) force as shown in Figure 1.

Hot Disk TPS 7 software was used to conduct the tests and analyze the obtained data. This software calculates the thermal conductivity using two methods:1) no assumptions about material properties are made, and 2) literature values for the volumetric specific heat (vCp) in MJ/m³K are used. All tests were calculated using method 2. Calibration was performed at the beginning of the day using the test protocol SIS2343 mild steel standards. The test protocol provided by Thermtest Instruments provides the standard results for thermal conductivity, thermal diffusivity, and heat capacitance, which were used to compare and prove that the system is functioning properly. Ten tests were performed for each material type at room temperature (between 20°C to 20.5°C), at -70°C, and at 200°C to obtain statistically relevant data. Samples were taken from the same material cylinder stock

described previously in the report. The samples were electro discharge machined (EDM) to a right cylinder that was 1 x 1 inch in diameter and height.

Table II: Experimental values of thermal conductivity and diffusivity at room temperature (20°C to 20.5°C)

Test #	SS 304L TC (W/mk)	SS 304L Thermal diffusivity (mm²/s)	Al. 6061 TC (W/mk)	Al. 6061 Thermal diffusivity (mm²/s)	OFHC TC (W/mk)	OFHC Thermal diffusivity (mm²/s)
1	14.25	3.581	174.5	72.76	393.9	114.5
2	14.25	3.58	176.1	72.60	376.8	109.5
3	14.25	3.581	175.7	72.93	378.1	109.9
4	14.25	3.582	176.5	72.62	473.7	137.7
5	14.25	3.581	175.8	74.35	393.7	114.5
6	14.26	3.583	179.9	72.32	405.5	117.9
7	14.25	3.58	175	71.82	435.1	126.5
8	14.25	3.581	173.8	73.00	443.7	129
9	14.25	3.58	176.7	73.17	387.6	112.7
10	14.33	3.601	177.1	72.14	382.5	111.2
Average	14.259	3.583	176.11	72.771	407.06	118.34
Stdev	0.024	0.006	1.590	0.655	31.080	9.040

Table II shows the measured thermal conductivity and diffusivity at room temperature from our work. The thermal conductivity for these materials were calculated using literature values of the volumetric specific heat were used for measuring the thermal conductivity of Stainless Steel 304, Aluminum 6061 and the OFHC Cu. The vCp values were calculated by using 3.98 MJ/m³K for SS304, 2.42 MJ/m³K for Aluminum 6061, and 3.44MJ/m³K for OFHC Cu, respectively. Room temperature tests were performed on the bench top as seen in Figure 1. Although the temperature varied from 17°C to 22°C for the room temperature tests for different days of testing, it was constant for each individual test on each material.

All -70°C, and at 200°C measurements were performed in a SPX model TUJR Environmental Test Chamber which has a temperature range of -70°C, and at 200°C shown in Figure 2.

To obtain a comparison reading from the chamber monitor thermocouple, which is read on the controller, a secondary type K thermocouple was placed on top of the samples and read external to the chamber on a hand-held Omega readout. For all -70°C, and 200°C tests, the secondary thermocouple was within 4°C of the target temperature. Results for -70°C, and at 200°C are shown in Tables III and IV, respectively. It is important to note that copper formed an oxide layer at 200°C, so the readings in this regime might not be reliable.

Table III: Experimental values of thermal conductivity and diffusivity at -70°C

Test #	SS 304L TC (W/mk)	SS 304L Thermal diffusivity (mm²/s)	Al. 6061 TC (W/mk)	Al. 6061 Thermal diffusivity (mm²/s)	OFHC TC (W/mk)	OFHC Thermal diffusivity (mm²/s)
1	11.74	2.949	126	52.05	363.7	105.7
2	11.77	2.957	132.4	54.71	360.4	104.8
3	11.75	2.952	125.2	51.74	362.8	105.5
4	11.72	2.945	132	54.54	360.2	104.7
5	11.7	2.939	121.6	50.25	358.6	104.2
6	11.71	2.942	125.5	51.84	354	102.9
7	11.72	2.945	131.2	54.23	358.6	104.3
8	11.67	2.931	127.1	52.51	356.4	103.6
9	11.72	2.944	132.3	54.67	360.4	104.8
10	11.66	2.929	134.4	55.53	360.2	104.7
Average	11.716	2.943	128.8	53.207	359.53	104.5
Stdev	0.032	0.008	3.987	1.650	2.694	0.787

Table IV: Experimental values of thermal conductivity and diffusivity at 200°C

Test #	SS 304 TC (W/mk)	SS 304 Thermal diffusivity (mm²/s)	Al. 6061 TC (W/mk)	Al. 6061 Thermal diffusivity (mm²/s)	OFHC TC (W/mk)	OFHC Thermal diffusivity (mm²/s)
1	16.41	4.123	170.7	70.55	347	100.9
2	16.87	4.239	184.4	76.21	343.5	99.87
3	16.99	4.268	183.8	75.93	361.1	105.
4	17.00	4.272	180.9	74.77	341.3	99.2
5	17.12	4.203	178.7	73.83	359.5	104.5
6	17.00	4.271	182.8	75.54	349.4	101.6
7	17.14	4.307	174	71.92	383.8	111.6
8	17.27	4.339	194.6	80.41	349.4	101.6
9	17.09	4.295	177.4	73.3	343.5	99.86
10	17.55	4.409	181.3	74.94	350.6	101.9
Average	17.04	4.272	180.86	74.74	352.91	102.6
Stdev	0.28	0.072	6.171	2.55	12.006	3.496

The following table shows measured values at the temperatures used for conductivity measurements and summarizes the average measured values from Table II, III and IV. The measured values are compared with literature values obtained from the NIST/CRC Properties of Selected Materials at Cryogenic Temperatures [3]

Table IV: Table showing measured thermal conductivities and literature values

Temperature (K,C)	SS304L TC (W/mk)	NIST SS304L TC (W/mk)	Al6061 TC (W/mk)	NIST Al6061 TC (W/mk)	OFHC TC (W/mk)	NIST OFHC TC (W/mk)
203,-70	11.716	12.72	128.770	136.81	359.530	399.79
293,20	14.259	15.12	176.110	154.35	407.060	392.81
473,200	17.044	19.55	180.860	167.69	352.910	383.41

The slight variations in the current and the reported literature values could be due to 1) pedigree of the materials involved, 2) assumptions used to calculate the final thermal conductivity and 3) the measurement technique used. Figure 1 shows the variation of the average thermal conductivity as a function of temperature. It can be seen that the variations in thermal conductivity are insignificant. This is consistent with expected and literature results. Additionally, the trends of the measured data correspond with the trends from the NIST database [3]. In general, for pure metals the thermal conductivity is due to the free electrons and is directly proportional to the absolute temperature (in K) and electrical conductivity. However, electrical conductivity decreases as the temperature is increased. Hence, the thermal conductivity should exhibit only minor increases or decreases in the ranges measured in this report, which was indeed observed. However, as the temperature approaches absolute 0, the conductivity decreases rapidly. Many pure metals have a peak in thermal conductivity between 2 and 10 K.

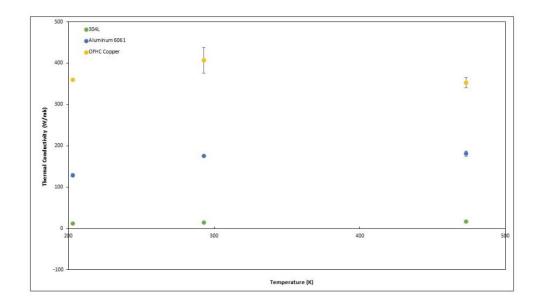


Figure 1. Average thermal conductivity as a function of temperature. Average values from Tables II, III and IV were used in this plot along with the calculated standard deviations.

2. Hardness measurements

A Struers DURASCAN 70 hardness testing machine was used to obtain Vickers hardness for the stainless steel 304, aluminum 6061, and OFHC Cu. The discs were EDM to 0.125 x 1 inch and were obtained from the same material stock as mentioned in the Specimen preparation for measurements section above.

To avoid artifacts in the measurements from surface topography, the discs were additionally polished using 0.04 micron colloidal silica. The Vickers hardness values were measured using a test load of 1 kg for Stainless Steel 304, 0.5 kg for both Aluminum 6061 and Cu, with 10 measurements per sample that were randomly distributed. The average hardness (MPa) for each material is shown in Table V. MatWeb Material Property reports the following values for Vickers Hardness for the three materials, SS304L (129 MPa) [4], Aluminum 6061 (107 MPa) [4], and OFHC copper (50-90 MPa) [5]. The differences in the reported and measured values could be attributed to the exact material microstructure. The higher hardness values here could be attributed to material impurities and also variation in the grain size as compared to the reported data.

Table V: Experimental values of Vickers Hardness measurements (MPa)

Test #	SS 304L	Al 6061	OFHC
1	252	121	100
2	285	112	96.7
3	296	114	94.7
4	275	109	95.5
5	272	115	100
6	300	126	88.9
7	282	117	91.3
8	263	117	90.1
9	302	126	84.7
10	282	114	95.2
Average	280.9	117.1	93.71
Stdev	15.25	5.37	4.67

References:

[1] https://www.hotdiskinstruments.com/products-services/sensors/kapton-sensors/

- [2] https://www.ors.org/Transactions/59/PS2--085/1535.html
- [3] Bradley, P. and Radebaugh, R. (2013), Properties of Selected Materials at Cryogenic Temperatures, CRC Press, Boca Raton, FL, [online], https://tsapps.nist.gov/publication/get_pdf.cfm?pub_id=913059 (Accessed September 26, 2021)
- [4]http://www.matweb.com/search/datasheet_print.aspx?matguid=1b8c06d0ca7c456694c7777d9e 10be5b
- [5] http://www.goodfellow.com/A/OFHC-Copper.html